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Contacting Single Template Synthesized Nanowires for Electric Measurements

A. Bachtold, C. Terrier, M. Krüger, M. Henny, T. Hoss, C. Strunk, R. Huber, H. Birk, U. Staufer, and C. Schönenberger [a] *

[a]Institute of Physics, University of Basel, Klingelbergstrasse 82, CH-4056 Basel, Switzerland

With template synthesis nanowires with diameters as small as 5 nm can be fabricated using electrochemical plating in nanopores. In this work contacts are fabricated enabling electrical measurements on one nanowire. A combination of chemical methods and e-beam lithography is used. The successful contacting is demonstrated for the case of Ni wires.

1. INTRODUCTION

Template synthesis is a chemical method alternative to conventional lithography which enables the fabrication of ultra-narrow nanowires with diameters down to $\lesssim 5$ nm [1]. Using electron-beam (e-beam) lithography in combination with lift-off, which is the conventional method for the fabrication of nanostructures, metallic wires with diameters in the range of $\approx 10 \ldots 50$ nm can be obtained [2]. This approach is possible for materials which can be deposited by evaporation or sputtering. It has not yet been explored in case of conducting polymers. However, nanowires of this material have already successfully been realized with template synthesis [3]. Template synthesis starts off with a template that contains an array of (nearly) monodisperse nanopores. These pores are filled in solution by electrochemical plating which results in an array of nanowires. Other alternatives to define polymeric nanowires are to locally induce polymerization by e-beam [4] or (although only for somewhat larger wires) to structure the wire from a polymeric film by a subtractive method. With regard to the latter, however, conducting polymers are known to be very sensitive to environmental conditions, e.g. humidity, and are subject to a relative rapid aging if left unprotected in air. Hence, any etching process following polymerization and doping should be avoided. Furthermore, it is desirable to have the conducting polymer encapsulated for environmental protection. This is guaranteed in case of template synthesis where the formed wires are embedded in the template. The most commonly used templates are porous alumina membranes [5] and commercially available screen filters (e.g. from Nuclepore and Poretics) which are thin polycarbonate foils containing pores obtained from etching nuclear damage tracks [6]. Typical templates have a high density of pores, $\approx 10^8 - 10^{10}\,\mathrm{cm}^{-2}$. This allows the fabrication of large assemblies of nearly identical nanowires, ideal for measuring magnetic [7] and optical [8] properties. In contrast to these measurements, where the limited measuring sensitivity demands a large assembly, electric measurements would best be done on one single nanowire. With regard to electric properties, template-synthesis has proven to be a powerful method: high aspect-ratio nanowires consisting of periodically stacked magnetic multilayers have been investigated [9]. Cai and Martin [3], on the other hand, have reported a spectacular size dependence for template-synthesized conducting polymers. In either case, a reliable method that allows to electrically address one single nanowires is lacking.

In this paper an electric contacting scheme is outlined and its success is demonstrated for Ni wires grown in polycarbonate templates with a nominal pore diameter $d_N = 80$ nm, membrane thickness $t = 6\,\mu$m, and mean pore length of

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6.5 μm. The mean pore length is $> t$ because of an angular spread of the pore axis of $\pm 30°$.

2. FABRICATION

Due to the high density of pores, which on average are a distance of 300 nm apart, contacting one nanopore asks for high resolution lithography. As mentioned before, conducting polymers are sensitive to environmental conditions and should certainly not be exposed to developer. With this in mind, our process has been designed so that the final nanowire is grown in the last step subsequent to *all* lithographic processes. With reference to the schematic in Fig. 1, the process is outlined in the following. A gold layer, thick enough to close the pores, is deposited on one side of the membrane (typically a piece 1 cm in diameter). This layer serves as the bottom electrode for all electrodeposition steps as well as for electric measurements. Then, Ni is electrochemically deposited into all pores. The growth is stopped at the instant when spherical Ni caps start to nucleate on top of the membrane [10]. This initial filling protects the pores from all lithographic processes that follow. To make spin coating possible, the membrane is permanently fixed onto a rigid substrate with the Au layer facing the substrate. Then a resist layer is spin-coated on top of the membrane. A positive Novolak-based photoresist was selected instead of PMMA, because of its higher chemical resistance. Using e-beam lithography, a contact hole (≈ 500 nm in diameter) is opened in the resist selecting one or a few pores. Similarly to the above mentioned protection of the pores, electrochemically plated Ni is also used to fill the contact hole for protection (not shown in Fig. 1). To complete the process a top electric contact pad has to be defined. This is done as follows: a homogeneous Au layer of thickness 100 nm is evaporated over the whole sample. Then, a second positive resist (PMMA-MA copolymer) is spin-coated and structured with e-beam. The structure consists of a) an opening of size $\approx 10\,\mu$m centered above the previously made contact hole, and b) open lines separating the central area (several 100 μm) from the rest. The parts of the Au layer which are exposed through the openings of the structured PMMA-MA mask are now removed by chemical wet-etching using KI. Finally, the PMMA-MA resist is stripped off completing the structure.

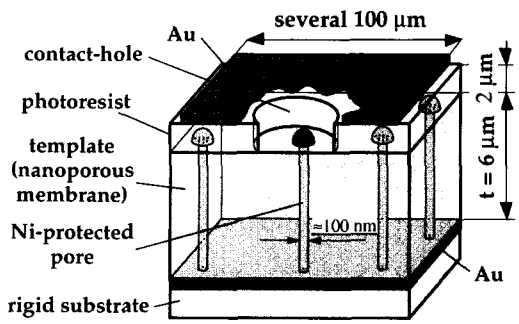

Figure 1. Schematic (not to scale) showing a completely structured device before the dissolution of the Ni in the pore selected by the contact hole.

A very important aspect of the outlined process is the protection of the pores which is maintained during all processing! Due to the permanent protection, the devices may be stored at this stage for weeks until a final nanowire, a conducting polymeric nanowire, for example, is required for measurements. Then, the Ni protection is electrochemically removed through the contact hole using sulphuric acid. Next, the opened pores are refilled with the desired material. This time, however, the electrochemical growth is continued until the pore and the contact hole are filled as well as the gap between the contact hole and the Au pad, see Fig. 2. An important aspect for the reliable filling of the pores in our scheme is the fact that the opened pores are automatically wetted by an aqueous solution due to the preceeding dissolution of the Ni (wet etching), which proceds from the top contact down to the bottom of the pore. This is very advantageous, since electrochemical plating into dried pores can be quite unreliable due to wetting problems. Fig. 2 serves to illustrate the step, in which the final electrically

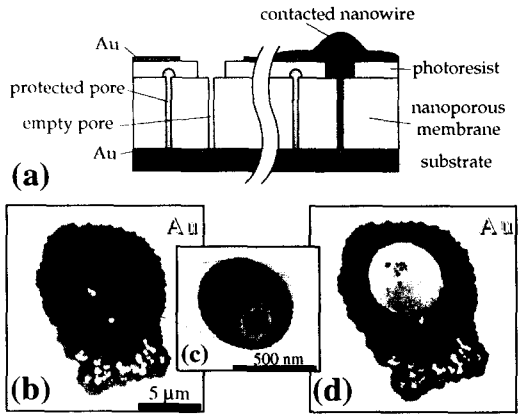

Figure 2. (a) shows one selected pore with dissolved Ni protection on the left and on the right refilled with the desired material forming electric contact to the top Au layer. The SEM images are top views of one and the same device obtained after dissolution of the Ni (b,c) and after (partial) refilling (d).

contacted nanowire is formed. The left part of a) shows one pore after dissolution of the Ni used for protection before. In the right part, the previously opened pore has now electrochemically been refilled with the material of interest. The filling is stopped once the material has established contact at the gold pad. The three SEM snapshots, b)-d), have been taken for one and the same device: b) and c) after the dissolution of the Ni protection, and d) after refilling the pore (in this case using also Ni). The two figures b) and c) nicely illustrate the hierarchy of three levels of holes: the nanopore of the template can be seen in c) within the contact hole of size 500 nm. This contact hole is seen in b) to be located within a larger opening in the Au layer of $\approx 10\,\mu$m. The ragged border of this hole is due to unavoidable underetching in the Au-etch step. Fig. 2d) demonstrates that the same pore can sucessfully be refilled in the final process. The bright 'blob' centered above the contact hole is freshly plated Ni that has filled the pore and grew out of the contact hole. For visualization purposes, the plating was stopped before the Ni could make contact with the Au surrounding.

3. ELECTRIC MEASUREMENTS

It is our goal to measure the intrinsic electric resistance of nanowires formed by template synthesis. For this to be possible, we need to know the number of wires which are contacted. Since the nanopores are randomly distributed on the surface of the polycarbonate membrane, the number of pores being selected by the arbitrarily placed contact hole in the photoresist changes for every device (from zero to a few wires). In order evalute this number, a statistical analysis of the measured conductance G is used. This is demonstrated in the following. In Fig. 3a the conduc-

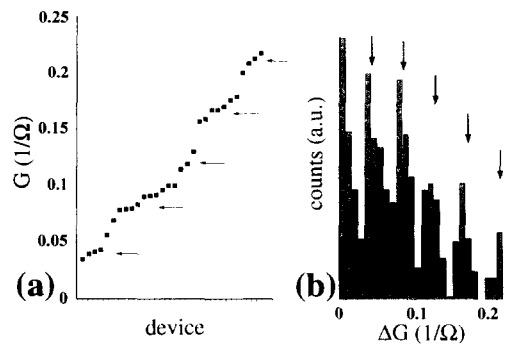

Figure 3. Left: Conductance values of 30 devices with Ni wires represented in acending order. Right: Histogram for the difference in conductance of different devices.

tances G, measured at $T = 300$ K, for 30 identically prepared devices consisting of pores filled with Ni are shown. Each solid dot corresponds to one device. The horizontal axis, the device number, has been sorted such that the G-values appear in ascending order. If all pores were exactly

identical, sharp steps would appear. But there is a clear accumulation seen, in particular around G-values of 0.042, 0.084, and $0.168\,\Omega^{-1}$ (arrows). Since there are no conducting devices found with $G \leq 0.03\,\mathrm{S}$, the smallest conductance of $\approx 0.042\,\mathrm{S}$ has to correspond to one single nanowire. This 'unit' of conductance can more accurately be derived from the histogram in Fig. 3b representing the difference in conductance ΔG for two different devices taking all possible cominations. This histogram shows pronounced equidistantly spaced peaks, the distance of which is a measure of the mean conductance for one single nanowire. For the mean resistance $R = 1/G$ a value of $R = 23\,\Omega$ is deduced. Using the measured resistivity of $\rho = 7.7\,\mu\Omega\,\mathrm{cm}$ for electrodeposited Ni films, the average inner pore diameter of 160 nm [10], and the mean pore length of $6.5\,\mu\mathrm{m}$, a single Ni wire should have an average resistance of $25\,\Omega$ in excellent agreement with the present experiment. This result also proves that contact resistances can be neglected.

Finally, Fig. 4 shows a first magnetoresistance measurement, demonstrating that the nanowires – contacted according to the description given above – can be used for electric transport experiments at low temperatures.

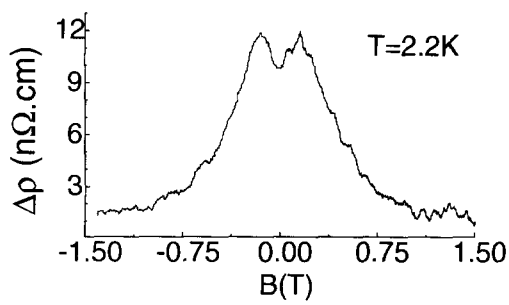

Figure 4. Magnetoresistance of 4 simultaneously contacted Ni nanowires in parallel measured at $T = 2.2$ with the magnetic field B directed $\perp$ to the Ni nanowire.

4. CONCLUSION

Conducting nanowires are electrochemically fabricated in templates consisting of a large and dense array of nanopores. Electric contacts to one or a few nanowires have successfully been made using a newly developed multilevel process. First electric measurements on a set of Ni nanowires demonstrate that the conductance of one single nanowire can be determined if a statistical analysis is applied. After this analysis, the devices with only one contacted Ni wire may further be used, for example, for conducting polymer wires. This will be worked on in the future.

REFERENCES

1. C. R. Martin, Science 266 (1994) 1961.
2. H.I. Smith and H.G. Craighead, Physics Today, February (1990) 24; special issue on *Nanotechnology*, P. Vettiger, U. Staufer, and D.P. Kern (eds.), Microelectronic Eng. 32, September issue (1996).
3. Z. Cai, C.R. Martin, J. Am. Chem. Soc., 111, (1989) 4138.
4. S.H.M. Persson, P. Dyreklev, and O. Inganäs, Adv. Mater. 8 (1996) 405.
5. R.C. Furneaux, W.R. Rigby, and A.P. Davidson, Nature 337 (1989) 147.
6. R.L. Fleischer, P.B. Price, and R.M. Walker *Nuclear Tracks in Solids*, Univ. of California Press, Berkley, 1975.
7. A.ALMawlawi, N.Coombs, and M.Moskovits, J. Appl. Phys. 70 (1991) 4421; T.M. Whitney, J.S. Jiang, P.C. Searson, and C.L. Chien, Science 261 (1993) 1361; J. Meier, B. Doudin, J.-Ph. Ansermet, J. Appl. Phys. 79 (1996) 6010.
8. B.M.I. van der Zande, M.R. Böhmer, L.G.J. Fokkink, and C. Schönenberger, J. Phys. Chem. B 101 (1997) 852.
9. L. Piraux et al. and A. Blondel et al, Appl. Phys. Lett. 65 (1994) 2484 and 3019, respectively.
10. C. Schönenberger, B. van der Zande, L. Fokkink, M. Henny, C. Schmid, M. Krüger, A. Bachtold, R. Huber, H. Birk, U. Staufer, J. Phys. Chem. 101 (1997) 5597.